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Properties of Catalysts Made of Metal Cuprates

937D0142B Moscow ZHURNAL FIZICHESKOY
KHIMII in Russian Vol 67 No 8, Aug 93 (manuscript
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[Article by L. Forni, C. Oliva, and F.P. Vatti, University of Milan/ ITALY, N.A. Sinityna, S.V. Sorochkin, A.V. Moyev, and A.V. Vishnyakov, Russian University of Chemical Technology imeni D.I. Mendeleyev, Moscow; UDC 541.128]

[Abstract] An experimental study of calcium and strontium cuprates was made concerning their possible use as catalysts for soft oxidation of toluene in benzaldehyde and for dissociation of nitric oxide in a nitrogen atmosphere. Four such compounds were selected: $\text{SrCuO}_{2+\delta}$ (A), $\text{Ca}_2\text{CuO}_{3+\delta}$ (B), $\text{Sr}_3\text{Cu}_5\text{O}_{8+\delta}$ (C), $\text{Ca}_{1.65}\text{Sr}_{1.35}\text{Cu}_5\text{O}_{8+\delta}$ (D). Specimens of each were produced by the sol-gel process: mixing nitrates of the metals (Ca, Sr, Cu) with 30% aqueous solution of polyacrylic acid (M=50,000) - drying the gel mass at 473-523 K temperatures in air and then calcining at 773-973 K temperatures in air - final 50 h long heat treatment at 1143 K either under vacuum (residual pressure 10 Pa) or in air. Chemical analysis of the specimens by iodometric titration revealed a nonstoichiometric oxygen content, the deviations being: $d=0.1$ (compound A), $d=0.01$ (compound B), $d=0.15$ (compound C), $d=0.4$ (compound D). Examination of the materials included morphological analysis of the powders under a Cambridge Stereoscanner 150 scanning electron microscope, x-ray phase analysis of solid specimens in a Siemens D-500 diffractometer with a CuK_α -radiation source and a Ni-filter, measurement of cation surface concentrations with an ERMA LINK model 860 instrument, and porosity measurement by the nitrogen sorption-desorption method in a SOPROMATIC 1800 ERBA apparatus at liquid-nitrogen temperature. Sorption, desorption, and catalytic activity for toluene oxidation in benzaldehyde and for NO dissociation in nitrogen were measured in TPD-TPR experiments with an injection reactor, using helium as the carrier gas and a quadrupole mass-spectrometer as detector. Presence of adsorbed radicals and the nature of paramagnetic centers, including copper, were revealed in EPR (electron-paramagnetic resonance) spectra recorded with a VARIAN E-Line Century Series spectrometer at various temperatures. The results indicate that only compound D is capable of adsorbing significant amounts of toluene, formation of aldehyde on its surface being facilitated by free active oxygen in the specimen. While most of the adsorbed toluene undergoes here complete combustion into CO , CO_2 , and H_2 , at least some toluene is being adsorbed by centers not capable of oxidizing it and readily desorbed from them upon injection of air (oxygen). Compounds C and D are good and selective catalysts for NO dissociation, compound D having a longer life. Compounds A and B are

poor catalysts for toluene oxidation and for NO dissociation. Figures 7; references 31.

Heterogeneous Chemiluminescence: Excitation Mechanisms, Part 1, Atomic Probe for Diagnostic Examination of Adsorbed Atoms

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KHIMII in Russian Vol 67 No 8, Aug 93 (manuscript
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[Article by V.P. Grankin, N.D. Grankina, and V.V. Styrov, Mariupol Institute of Metallurgy, UDC 535.377:541.128]

[Abstract] An apparatus has been built for examining, with an atomic probe, the coverage of surfaces of luminescent solid substances by adsorbed atoms or radicals. This apparatus was tested on ZnS , ZnS-Tm , and CdS-Ag targets. Such a target was placed on top of an electric microfurnace and together with it slid into a hydrogen chamber having a wall of molybdenum glass with three holes: one for insertion of the target, one for evacuation of air, one for subsequent admission of hydrogen. The chamber had also two windows: one letting in light from a mercury lamp for photoexcitation of the target and one letting out light to a photomultiplier. Molecular hydrogen generated in a special electrolyzer was passed through a purifier into an electrodeless high-frequency discharge tube. A flux of hydrogen atoms generated here by dissociation of molecules proceeded into the chamber for the purpose of reacting with target surface atoms and thus exciting heterogeneous chemiluminescence. The purifier, made of palladium, served also as a hydrogen flow regulator. The hydrogen pressure in the discharge tube, dependent on the purifier temperature, was regulated indirectly by means of an electric heater with current control which regulated that temperature. Hydrogen atoms generated by any of two or both pyrolyzers, electrically heated tungsten coils, were injected into the chamber for the purpose of probing the target surface. Measurements could be made with reacting hydrogen atoms from the discharge tube and probing hydrogen atoms from the pyrolyzers as well as hydrogen molecules at the target surface. The relative magnitude of the hydrogen fluxes were: $j_1 + j_2$ (probing hydrogen atoms from both pyrolyzers) $\leq 0.001j_r$ (reacting hydrogen atoms from discharge tube) $\leq 0.05j_m$ (hydrogen molecules). Tests were performed at a 320 K temperature under a 0.1 torr pressure of molecular hydrogen. The concentration of hydrogen atoms on the target surface was measured sufficiently fast, in less than 5 s, for the surface coverage by hydrogen molecules not to change significantly. The flux of probing hydrogen atoms (atomic probe) was generated and injected in pulses of 1-2 s duration. The intensity of heterogeneous chemiluminescence, proportional to the degree of surface coverage by hydrogen atoms, was indicated by the intensity of the flash of glow emission by the target

surface upon incidence of that atomic probing flux. These measurements revealed a dependence of the luminescence intensity on the length of target soaking time in molecular hydrogen. At a constant concentration of probing hydrogen atoms a change in that intensity was evidently caused by a change in the concentration of hydrogen atoms reacting with target surface atoms and its increase in time was possibly due to dissociative hydrogen adsorption by the target surface. Some tests were performed at 320 K and also 370 K temperatures under the same 0.1 torr pressure of molecular hydrogen, for measurements after the steady flux of reacting hydrogen atoms from the discharge tube had been cut off. In these tests the luminescence decayed rather fast to a weak afterglow. A theoretical analysis of the results leads to the conclusion that heterogeneous chemiluminescence of ZnS-Tm (ZnS, CdS-Ag) at a temperature below 350 K is excited essentially by the impact recombination mechanism and that annihilation of atoms on the target surface with an attendant long afterglow at a temperature above 350 K occurs essentially by the Langmuir-Hinshelwood diffusion mechanism of recombination, impact recombination not being possible in the absence of hydrogen atoms in the gaseous atmosphere. Figures 5; references 5.

Effect of Microalloying on High-Temperature Creep of Cu_{2-x}Se Alloys

[Article by V.N. Geminov, A.G. Bochvar, and A.G. Korzhuyev, Institute of Metallurgy imeni A.A. Baykov at Russian Academy of Sciences, Moscow; UDC 621.315.592:541.135]

[Abstract] An experimental study of Cu_{2-x}Se superionic semiconductor compounds with mixed electronic ("hole" p-type) and ionic (Cu^{++} ions) conductivities was made concerning the changes in their mechanical properties, plasticity and strength, upon addition of microquantities of II, III, IV, V-group metals. Up to 1 atom.% of Al, Zn, Cd, In, Sb were added, the selenides of these five metals being at equilibrium with Cu_2Se in the respective ternary systems. Alloying was done under vacuum in quartz tubes, the temperature being raised gradually to 1200°C within 3 h. The alloys were first maintained in their liquid state for 1 h and then cooled in air. Chemical equilibrium in the solid state was attained 200 h long annealing: alloys with In or Zn at 600°C and then at 400°, alloys with Sn or Al at 500°C, alloys with Sb at 400°C. Straight cylindrical test specimens 20 mm high and 7 mm in diameter were produced by compaction of the 50-100 mm powder fraction at room temperature under a pressure of 1 GPa. They were tested in compression under vacuum (residual pressure about 0.001 torr at $T = 673, 773, 873$ K temperatures, vertical constant loads of 4-40 MPa being successively applied for 100 h at each temperature. Dimensional changes were measured with a

cathetometer, the cylinders shrinking in height and expanding laterally into barrels. For prediction of their creep limit, the test data were plotted into $\sigma = f(\varepsilon)$ stress-strain curves referring to the mean-over-height diameter and these curves then converted into the $\sigma = f(P)$ basic parametric diagram where $P = T(16.6 - \log \frac{d_0}{d}) - L - K\sigma$ (here 16.6 representing the Debye oscillation period of atoms in the crystal lattice). This diagram closely approximates the experimentally obtained piecewise-linear $\sigma(P)$ characteristic of five specimens: two specimens of $\text{Cu}_{0.199}\text{Se}$, one specimen of $\text{Cu}_{0.662}\text{Zn}_{0.0033}\text{Se}_{0.3345}$, two specimens of $\text{Cu}_{0.64}\text{Sb}_{0.01}\text{Se}_{0.35}$. This confirms the universality of the basic parametric diagram and that, therefore, the class of doped Cu_{2-x} (Al, Zn, Cd, In, Sb) semiconductors do not depart from the general rule of plastic deformation: principally intragranular stressing at temperatures below 0.4T and intergranular stressing at temperatures above 0.4T_{melt}. With the aid of the basic parametric diagram, one therefore can not only extrapolate the strain rate test data along the time scale down to microhours (under high stress) and up to kilohours (under low stress) but also estimate the effect of microalloying on the useful life of the material: length of time before the strain exceeds a given limit under a given load) according to the relation $t/t^* = 10^{(P-P^*)/T}$ (P^*, t^* - characterizing unalloyed Cu_{2-x}). The effect of microalloying is thus found to be a decrease of plasticity and an increase of strength at high temperatures. The authors thank V.F. Bankin and N.N. Filipovich for synthesizing the materials, O.V. Goritskiy for assisting with the measurements. Figures 3; references 12.

Ionospheric Plasma's Interaction With Spacecraft Materials and Equipment

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OBRAZOTKI MATERIALOV in Russian No 3,
May-Jun 93 pp 40-47

[Article by A.I. Akishin, S.K. Guzhova, Scientific Research Institute of Nuclear Physics and Moscow State University imeni M.V. Lomonosov; UDC 537.311]

[Abstract] The effect of the processes on the surface of materials exposed to corpuscular flows of ionospheric plasma on the long-term functioning of spacecraft (KA) in low circumterrestrial orbits and the accuracy of on-board instrument readings is discussed, and it is noted that at an altitude of 150-500 km, the satellite's (ISZ) frontal surface is exposed to an approach flow of rarefied ionospheric plasma with a flux density, depending on altitude, of $10^6-10^{13} \text{ cm}^{-2}/\text{s}$ for neutral particles and $10^{12}-10^{11} \text{ cm}^{-2}/\text{s}$ for ions due to high orbital velocities of 7.8 km/s, leading to the development of a self-consistent potential. The interaction of ionospheric particles with the satellite's outside surface material is investigated in detail, and the dependence of the interacting atomic oxygen concentration and flux density on the orbital

altitude under various conditions, the altitude dependence of the spectral luminance intensity and N₂ and O concentration, the spectral luminance characteristic, and the dependence of the polyimide mass loss due to ablation on the oxygen atom energy are plotted. The findings demonstrate that the oxygen-containing plasma interaction with spacecraft materials in low orbits leads to a degradation of their physical properties and changes in the lower atmosphere's composition as well. It is speculated that the results fall far short of explaining all aspects of the problem; the need for subsequent full-scale and laboratory materials science research is emphasized. The findings obtained in the course of Space Shuttle experiments with Capton and Mylar films are utilized widely in the study. Figures 4; tables 2; references 26: 10 Russian, 16 Western.

Ionospheric Plasma's Interaction With Spacecraft Materials and Equipment

937D0148A Moscow FIZIKA I KHIMIYA
OBRABOTKI MATERIALOV in Russian
No 3, May-Jun 93 pp 40-47

[Article by A.I. Akishin, S.K. Guzhova, Scientific Research Institute of Nuclear Physics and Moscow State University imeni M.V. Lomonosov; UDC 537.311]

[Abstract] The effect of the processes on the surface of materials exposed to corpuscular flows of ionospheric plasma on the long-term functioning of spacecraft (KA) in low circumterrestrial orbits and the accuracy of on-board instrument readings is discussed, and it is noted that at an altitude of 150-500 km, the satellite's (ISZ) frontal surface is exposed to an approach flow of rarefied ionospheric plasma with a flux density, depending on altitude, of 10^6 - 10^{13} cm⁻²/s for neutral particles and 10^{12} - 10^{11} cm⁻²/s for ions due to high orbital velocities of 7.8 km/s, leading to the development of a self-consistent potential. The interaction of ionospheric particles with the satellite's outside surface material is investigated in detail, and the dependence of the interacting atomic oxygen concentration and flux density on the orbital altitude under various conditions, the altitude dependence of the spectral luminance intensity and N₂ and O concentration, the spectral luminance characteristic, and the dependence of the polyimide mass loss due to ablation on the oxygen atom energy are plotted. The findings demonstrate that the oxygen-containing plasma interaction with spacecraft materials in low orbits leads to a degradation of their physical properties and changes in the lower atmosphere's composition as well. It is speculated that the results fall far short of explaining all aspects of the problem; the need for subsequent full-scale and laboratory materials science research is emphasized. The findings obtained in the course of Space Shuttle experiments with Capton and Mylar films are utilized widely in the study. Figures 4; tables 2; references 26: 10 Russian, 16 Western.

Experimental Investigation Into Effect of Space Factors on Graphite Epoxy Composite Durability. 3. Effect of Gaseous Chlorine on Metallized Graphite Epoxy Composite State

937D0148B Moscow FIZIKA I KHIMIYA
OBRABOTKI MATERIALOV in Russian
No 3, May-Jun 93 pp 48-59

[Article by I.A. Kulikov, A.A. Kupriy, G.A. Yurlova, All-Union scientific Research Institute of Inorganic Materials imeni A.A. Bochvar, Moscow; UDC 541.15]

[Abstract] The decrease in the taut strained state concentration in graphite epoxy composite spacecraft panels due to the presence of microwave reflecting metallic layers and a reduction in the temperature differentials through the panel depth which, in turn, depends in the coat reflectance are discussed, and the interaction of a chemically active component—gaseous chlorine—with the metal coat intended for reflecting electromagnetic waves and removing static charges is investigated experimentally. The study is part 3 of a series, the previous two articles were published in 1992. Freshly prepared samples of graphite epoxy composite with a thin film coat (TPP) and samples exposed to moist aging at a 98% humidity for 90 days at room temperature are used in the study. The impact of chlorine liberated from the composite under the effect of such space factors as γ -irradiation, electron and proton flows, and, moisture, and aging on the metallized surface condition is examined. The types of thin film coats on the composite exposed to experimental factors and a comparison of various types of coat characteristics after γ - and electron irradiation and hydrostat exposure are summarized, and the infrared spectra of KMU-4L composites in the initial condition and after exposure, the behavior of chlorine liberation from the composite under various exposure conditions, and the effect of the exposure conditions and length of coated composites on the gaseous chlorine liberation are plotted. The study reveals that Invar foil lowers gaseous chlorine liberation much more than films while it does not display traces of corrosion. The authors are grateful to S.L. Zhdanov for helping with electron probe microanalysis of the film coat. Figures 3; tables 3; references 12: 11 Russian, 1 Western.

Low-Temperature Transformation and Magnetic Properties of Fe-B-Si Amorphous Strip

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[Article by G.N. Konygin, Ye.P. Yelsukov, V.A. Makarov, V.I. Ladyanov, and G.A. Sirotinina, Physical Technical Institute, Ural Department, Russian Academy of Sciences, Izhevsk, and Udmurt State University, Izhevsk; UDC 669.15'781'782:537.622]

[Abstract] A study examined the effect of annealing temperature and structural state on the formation of the magnetic properties (permeability and coercitive force) of amorphous alloys. An amorphous strip of $Fe_{80}B_{10}Si_{10}$ measuring 20 μm in thickness and 10 mm in width was used for the studies. The alloy was obtained by enriching iron with 2 atomic percent of the stable isotope ^{57}Fe to make it possible to obtain quality Mossbauer conversion electron spectra in a relatively short amount of time. The alloy strip was studied in its initial state and after cycles of isochronous annealing in a vacuum of about 10^{-3} Pa for 1 hour at temperatures of 50 to 800°C. The specimens were also studied by x-ray diffraction, Mossbauer absorption spectroscopy, and sliding-beam x-ray diffraction. X-ray crystallographic analysis of the $Fe_{80}B_{10}Si_{10}$ demonstrated that in its starting state, it is amorphous. The first signs of its crystallization, manifested in terms of the appearance of a weak intensity against the background of the "halo" that is characteristic of amorphous materials, were observed after annealing at temperatures of 295-315°C. As the annealing temperature was increased, the percentage of crystalline phase increased. The study $Fe_{80}B_{10}Si_{10}$ was found to undergo four stages of structural development: No structural changes were evident at 0-300°C. At 300-400°C, a body-centered crystal lattice with the characteristic close atomic order similar to that in the amorphous matrix developed; this process was accompanied by an explicit change in orientation of magnetic moments. At temperatures of 400-500°C, the percentage of body-centered crystal lattice increased until the amorphous matrix completely disappeared (after the specimen had been held for 1 hour at 455°C), and processes of close demixing began. At 500-800°C, a transition to an equilibrium phase diagram occurred. In its equilibrium state, the specimen consisted of two phases: a body-centered crystal lattice and Fe_2B . The Mossbauer spectroscopy studies pinpointed the temperature of onset of crystallization at around 250°C and indicated that the crystal regions lie at a depth of about 15 nm. The first of the above stages (i.e., 0 to 300°C) included three stages of structural relaxation. They were identified by changes in the specimen's magnetic characteristics at 75, 175, and 255°C. No structural changes were evident in the first two stages. In third stage, which is the most favorable from the standpoint of optimizing the magnetic properties of amorphous strips and increasing the stability of its properties over time, was accompanied by the appearance of nanocrystal

regions in the near-surface layer of the amorphous strip's contact side. Figures 7; references 10: 7 Russian, 3 Western.

Features of Microstructure and Magnetic Properties of High-Coercivity Microstrips of Alloys Based on Fe-Cr-Ni

937D0146B Yekaterinburg *FIZIKA METALLOV I METALLOVEDENIYE* in Russian Vol 75 No 2, Feb 93 (manuscript received 21 Apr 92; after revision 30 Sep 92) pp 69-75

[Article by A.R. Baraz, F.I. Stetsenko, A.M. Glezer, I.A. Komarova, and Yu.Ye. Chicherin, Central Ferrous Metallurgy Scientific Research Institute imeni I.P. Bardin; UDC 669.24'26:537.622]

[Abstract] A study examined the morphology, dimensions, and orientation of particles of the ferromagnetic phase in 7.5- to 15- μm -thick microstrips of the cold-deformed stainless steel alloys OKh12N12M3 and OKh12N12K3. The study specimens, which had been deformed by rolling to a degree of 98 percent with intervening cycles of tempering, were examined under light and transmission electron microscopes. The structural studies were performed on specimen strips with a thickness of 10 μm . The alloy OKh12N12M3 was determined to have a coercitive force of 29.5 kA/m, a saturation induction of 0.13, and a residual magnetization of 0.09. The alloy OKh12N12K3 was determined to have a coercitive force of 62.9 kA/m, a saturation induction of 0.15, and a residual magnetization of 0.13. The particles of the specimens' α -phase were found to have very diverse shapes and sizes and to contain the following characteristic types of particles/regions: 1) isolated small equiaxial or anisotropically shaped α -phase particles with a cross section of 4 to 10 nm and an axis (l/d) ratio of $<3:1$; 2) highly fragmented (i.e., in a block structure) particles of rather diverse shape with cross sections of 10 to 165 nm; and 3) colonies of elongated uniaxial particles with cross sections of 4 to 20 nm and an axis ratio between 3:1 and 12:1 (the direction of their long axes was generally close to the direction of rolling, and the particles often appeared to consist of individual fragments or chains of smaller particles within the confines of the defect band); and 4) regions with a relatively low density of α -phase measuring 0.5-3 μm . The studies indicated that the main contributor to the study alloys' level of magnetic properties are anisotropic particles with a heterogeneous structure consisting of disoriented fragments (blocks) and/or individual microparticles separated by γ -phase interlayers. Figures 4, table 1; references 17: 7 Russian, 10 Western.

Luminescence of Organic Dyes Adsorbed on Surface of Modified TiO_2 Films

937D0142C Moscow ZHURNAL FIZICHESKOY KHMII in Russian Vol 67 No 8, Aug 93 (manuscript received 24 Jun 92) pp 1665-1668

[Article by N.I. Kuntsevich, V.P. Poroshkov, and Ye.I. Vasilevskaya, Belarusian State University, Minsk; UDC 535.37]

[Abstract] An experimental study of TiO_2 films modified by basic compounds and of the luminescence of organic dyes adsorbed by them was made, considering possible use of organic dyes as indicators of the surface condition and of modifier participation in photographic processes. Hydrolytic TiO_2 films were tested with six modifiers enhancing their sensitivity during the development process: 1) p-phenylenediamine, 2) n,n-dimethyl-p-phenylenediamine, 3) n,n-dimethyl-p-phenylenediamine dihydrochloride, 4) triethylamine, 5) p-phenylenediamine tetraacetate, 6) p-phenylenediamine nucleus with COOH groups and $AgNO_3$. Some films were, prior to modification, soaked in air at temperatures within the 450-500°C for 1 h so as to transform them into films of polycrystalline anatase. Examination

of the films was done with four luminescent dyes: 2,2-difluoro-4,4(4'-dimethylaminostyrene)-naphto-[2,1-e]-1,2,3-dioxaborin (DB) sensitizing the films to visible light and the other three (rhodamine 6Zh, fluoroexon, Zn-octaethyl porphine) featuring a high fluorescence quantum yield. Following adsorption of the dyes from 0.001 mol/l solutions in methylene chloride, acetone, toluene, ethanol, or water, their optical absorption spectra and fluorescence spectra were measured with a Beckman UV-5270 spectrophotometer and with Fica-55 spectrofluorometers respectively. Their fluorescence spectra revealed a shift of the maximum-intensity wavelength from the intrinsic one within the fluorescence band, this shift being linearly dependent on the excitation wavelength. The results indicate that the amount by which the energy of electronic states in the dyes DB and Zn-octaethyl changes does not significantly depend on the nature of the modifier with which they interact (ΔE_{DB} varying only within the 0.048-0.088 eV range), which makes both dyes suitable for surface examination of TiO_2 films. The other two dyes rhodamine 6Zh and fluoroexon are not suitable for this, because their interaction with the film modifiers significantly alters their luminescence characteristics. Figures 1; tables 2; references 11.

Effect of Interface on Properties of Ti-W Fiber Composite Under Uniaxial Tension Conditions

937D0148I Moscow FIZIKA I KHIIMIYA OBRABOTKI MATERIALOV in Russian No 3, May-Jun 93 pp 156-159

[Article by V.I. Zhamnova, V.N. Meshcheryakov, Metallurgy Institute imeni A.A. Baykov at Russia's Academy of Sciences, Moscow; UDC 669.494:539.219]

[Abstract] The lack of sound scientific studies of titanium alloy-based fiber composites (VKM) prompted an investigation into the effect of the metal matrix/fiber interface in Ti-W fiber composites on the tensile stress behavior of the material. To this end, the constitution diagrams of the metallic systems are analyzed, showing that in Ti-W systems, the component interaction is limited to reciprocal dissolution while the resulting solid solutions have a high melting point. Plane samples of a fiber composite consisting of alternating layers of 0.35 and 0.30 μm dia. wire and matrix alloy foils with a wire fraction of 15-35% by weight are examined by X-ray spectral analyses under a scanning electron microscope and tested under uniaxial tension along the reinforcing wires until rupture at a 1,100 and 1,200°C temperature. A representative tensile strength curve of the material is plotted. The curve displays three stages—elastic deformation of both the matrix and the wire, elastic deformation of the wire and plastic flow of the matrix, and plastic flow of the matrix and wire until total composite failure. The findings demonstrate that such material can be regarded as promising from the viewpoint of thermodynamic compatibility of the components and from the viewpoint of forming an optimum interface condition. Figures 4; references 2: 1 Russian, 1 Western.

Distribution of Titanium in the Structure of Various Components of Superconducting Composites Based on Nb_3Sn

937D0146C Yekaterinburg FIZIKA METALLOV I METALLOVEDENIYE in Russian Vol 75 No 2, Feb 93 (manuscript received 15 Jul 92) pp 112-118

[Article by Ye.N. Popova, L.A. Rodionova, S.V. Sudareva, A.Ye. Vorobyeva, Ye.A. Dergunova, A.M. Chukin, A.K. Shikov, A.D. Nikulin, and Ye.P. Romanov, Metal Physics Institute, Ural Department, Russian Academy of Sciences, and All-Union Scientific Research Institute of Inorganic Materials imeni A.A. Bochvar; Moscow; UDC (621.763+539.945):620.186.1]

[Abstract] A study examined the distribution and effect of titanium in the structure of the various components of the composite Nb/Cu-Sn when it is used as an alloy-forming element in the amount of 0.2 percent by weight. The studies were performed on free bronze consisting of copper plus 13 weight percent tin and 0.2 weight percent titanium after homogenization at 700°C for 20 hours and

on multifilament composites consisting of Nb/Cu plus 13 weight percent Sn and 0.2 weight percent titanium (each composite contained 7,225 1-mm-diameter filaments) after diffusion annealing at 700°C for 72 hours. The fine structure of the composites and bronze were studied under a JEM-200 CX electron microscope, and the distribution of their elements was studied on a JCXA-733 scanning microscope/microanalyzer. The studies established that during the annealing process, the titanium diffuses from the bronze matrix through the Nb_3Sn layer into the niobium filaments. During the course of this process, the growth rate of the diffusion layer increased along with its thickness, and the anisotropy of the grains' shape decreased. No reduction in grain size was observed, however. The thermodynamics and kinetics of this redistribution of titanium in the composite were explained in terms of previously published mathematical studies of the partial free energy of titanium in solid solutions, the change in free energy during the face-centered crystal lattice → body-centered crystal lattice transition, and the condition of local equilibrium on the interface between solid solutions. The mechanism of the diffusion of titanium from the bronze into the filaments was shown to be more complicated but was also explained. Figures 7; references 14: 5 Russian, 9 Western.

Effect of Gallium Alloying on the Structure and Properties of a Superconducting Composite With Nb_2Sn

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[Article by Ye.N. Popova, L.A. Rodionova, S.V. Sudareva, N.V. Nikolayeva, A.K. Shikov, A.Ye. Vorobyeva, Ye.A. Dergunova, and A.M. Chukin, Metal Physics Institute, Ural Department, Russian Academy of Sciences, and All-Union Scientific Research Institute of Inorganic Materials imeni A.A. Bochvar; UDC (621.763+539.945):620.186.1]

[Abstract] A study examined the effect that alloying the bronze matrix of an Nb/Cu-Sn composite with gallium in the amount of 2 percent by weight has on the alloy's various components before and after diffusion annealing. The studies were performed on free bronze consisting of copper plus 11 weight percent tin and 2 weight percent gallium after homogenization at 700°C for 20 hours, as well as on a single filament of Nb/Cu plus 11 weight percent tin and 2 weight percent gallium after hot compaction and on a composite consisting of 7,225 filaments of the same composite that had been subjected to diffusion annealing at 700°C for 72 hours. The study specimens were examined under a JEM-200 CX transmission electron microscope and JCXA-733 scanning electron microscope/microanalyzer. The

addition of gallium to the study composites was demonstrated to cause an increase in the number of twins and ϵ -phase formations in the bronze matrix due to a decrease in packing defect formation energy. During the course of diffusion annealing, the gallium diffused into the Nb_3Sn layer and was partially preserved in the bronze matrix. Gallium was also found to increase the grain size in the superconducting layer and to not facilitate any increase in its thickness. The dissolution

of gallium in Nb_3Sn in large quantity during the course of diffusion annealing was found to result in an increase in the electrical resistance of the said phase in its normal state and, consequently, in an elevation of the upper critical field. This result is in turn associated with the higher current-conducting capability of gallium-alloyed conductors in high magnetic fields versus that of their nonalloyed counterparts. Figures 8; references 10: 4 Russian, 6 Western.

Experimental Study Concerning Effect of Cosmic-Space Factors on Durability of Carbon-Plastic Materials, Part 4: Laws Governing Accumulation of Corrosion Products from Metallized Carbon-Plastic

937D0143C Moscow *FIZIKA I KHIIMIYA OBRABOTKI MATERIALOV* in Russian No 4, Jul-Aug 93 (manuscript received 19 Jun 92) pp 38-48

[Article by I.A. Kulikov, A.A. Kupriy, and G.A. Yurlova, All-Russian Scientific Research Institute of Inorganic Materials imeni A.A. Bochvar, Moscow; UDC 541.15]

[Abstract] An experimental study was made concerning accumulation of corrosion products from a variously metallized carbon-plastic composite material in various environments without and with exposure to ionizing cosmic γ -rays, concern being the dependence of the corrosion kinetics on the irradiation dose and power as well as on the kind of environment and on the length of time the target material had been in it. The material in this experiment was the KMU-4L composite consisting of epoxy binder and carbon reinforcement fibers with either Invar foil cladding or four different thin-film metal coatings (Ti-Ni-Cu, Ti-S.S.-Cu; Ti-Ni-Al, Ti-S.S.-Al; S.S.= Cr18Ni19Ti stainless steel), the coatings having been deposited by the vacuum ion-plasma process. Specimens of each variant were tested: 1. in "dry" air (pressure 1 torr, temperature 20 \pm 1°C thermostatically controlled, humidity 30 \pm 10%); 2. under vacuum (pressure 0.1-1.0 mtorr, temperature 20 \pm 1°C); 3. in an inert atmosphere (argon pressure 1 torr, temperature 20 \pm 2°C); 4. in humid air (air pressure 1 \pm 0.1 torr, temperature 20 \pm 3°C, humidity 95-98% hydrostatically controlled). They were tested for emanation of gaseous chlorine and formation of Ni,Fe chlorides and Cu,Al carbonates over a period of 96,000 h. Accumulation of corrosion products in terms of Cl⁻, HCO₃⁻, and [Cu,Al,Fe] ions was measured after 50, 150, 250, and 400 days. In each environment all specimens were exposed to γ -rays in 1 MgR and 5 MgR doses, each applied at rates of 0.15 MgR/s, 0.5 MgR/s, and 1.5 MgR/s. Analysis of the corrosion products by spectroscopy and gas chromatography revealed no appreciable decomposition of Invar foil but an appreciable decomposition of thin-film metal coatings in the presence of γ -rays. The results of tests reveal, moreover, that: 1) irradiation of Invar-clad composite causes an accumulation of Fe⁺⁺ and Cl⁻ ions which increases with longer soaking time and with longer exposure time (larger irradiation dose), Moessbauer and NGR (nuclear-gamma resonance) spectroscopy of the Invar foil indicating a [Fe]/[Cl]= 1:2 ratio and thus formation of FeCl₂ not earlier than after about 250 days; 2) irradiation of Ti-Ni-Cu-coated composite causes an accumulation of Cu⁺, Cu⁺⁺, Cl⁻, HCO₃⁻, and also OH⁻ ions which increases with longer soaking time and with longer exposure time (larger radiation dose), up to 10 times

more gaseous chlorine emanating from copper-coated composite than from Invar-clad and HCO₃⁻ ions forming upon adsorption of CO₂ from the air; 3) irradiation of Ti-Ni-Al-coated composite causes an accumulation of Al⁺⁺⁺, Cl⁻, OH⁻, and HCO₃⁻ ions which is 2-2.5 times smaller than from copper-coated composite but 5 times larger than from Invar-clad composite; 4) lowering the radiation power reduces the overall accumulation of corrosion products within the same length of soaking time and increases the fraction of carbonates in thin-film coatings. Figures 4; Tables 4; references 2.

Experimental Study Concerning Effect of Cosmic-Space Factors on Durability of Carbon-Plastics, Part 5: Low-Energy Electrons, X-Rays, Ultraviolet Light

937D0143D Moscow *FIZIKA I KHIIMIYA OBRABOTKI MATERIALOV* in Russian No 4, Jul-Aug 93 (manuscript received 9 Jun 92) pp 49-64

[Article by G.A. Yurlova, M.Ya. Melnikov, and V.S. Aryutynov, Moscow State University, Department of Chemistry; UDC 541.15]

[Abstract] An experimental study was made concerning the effect of cosmic x-rays, ultraviolet light, and low-energy electrons on a carbon-plastic composite material: KMU-4L composite consisting of epoxy binder and carbon reinforcement fibers, flat and tubular specimens with a 0-90-90-0-90-90-0 reinforcement pattern. The x-ray tests were performed with a 65 kV tube covering the 2.50-0.186 γ x-ray and γ -ray spectrum and an FeSO₄ · 7H₂O dosimeter serving as detector. The wavelength was determined on the basis of x-ray absorption by aluminum plates, the effective mass coefficient of absorption μ/ρ (ρ - density of aluminum) being calculated as $\mu/\rho = [\log(p/p_0)]$ of aluminum plate; p, p_0 - power of absorbed dose in presence and absence of aluminum plates). The total absorbed dose varied over the 0.5-5 Mrad range and its power was approximately 83 rad/s. Four gaseous products of radiolysis were identified and quantitatively determined: hydrogen and carbon monoxide in a ChT-2M chromatograph (with a heat of combustion detector-katharometer, a 2.5 m high and 4.5 mm in diameter column of ASK silica gel, air as carrier gas flowing at a 45 ml/min rate); methane and ethane in a LChM-8M chromatograph (with a flame-ionization detector, a 2.5 m high and 4.5 mm in diameter column of KSK silica gel at a 30°C temperature, nitrogen as carrier gas flowing at a 45 ml/min rate). The specimens in this experiment weighed 0.2-0.4 g. The ultraviolet tests were performed with a DKSSh-1000 xenon lamp, its continuous emission spectrum approximating the solar one. They were performed on moist KMU-4L with various ratios of total surface area to area of lateral surfaces, after one year of room storage. The total absorbed dose of 253.7 nm light amounted to 2×10^{21} ev/cm². Only three

gaseous products of photolysis were identified and quantitatively determined in a ROMS-1 mass-spectrometer: hydrogen, carbon monoxide, methane. The electron bombardment tests were performed with 10-20 keV electrons (25 kV, 0.1 mA) in a special chamber under a pressure of 0.35-38 mtorr first and with the pressure then varied over the 0.58-3-10 mtorr range, with. They were performed on KMU-4L specimens after one year of room storage, on two kinds of them: 1) with a longitudinal lay of upper reinforcement fibers, after prior 100 heat treatment cycles; 2) with a transverse lay of upper reinforcement fibers, after prior 200 heat treatment cycles. The heat treatment consisted of following temperature cycles: 100°C for 15 min to room temperature for 10 min to -196°C for 15 min to room temperature for 10 min. Some specimens were not heat treated but only thoroughly dried before testing. Electron bombardment was followed by surface examination under an electron microscope and chromatographic analysis of gaseous

decomposition products. After the tests all specimens were tested in a micrometer for thickness and by the acoustic method (speed of ultrasound pulse propagation) for strength characteristics: shear modulus, bulk modulus, Young's modulus, and Poisson's ratio. The results indicate that: x-rays, ultra-violet light, and low-energy electron bombardment may cause not only decomposition of this carbon-plastic material with attendant emanation of gases but also additional aging (self-hardening) of the epoxy binder with an attendant formation of transverse bonds, which makes the magnitude of their destructive effect on this material less than linearly dependent on the dose. The combined effect of prolonged low-energy electron bombardment and prior cyclic heat treatment of moist material is probably be controllable by design of the surface reinforcement pattern. The authors thank Ye.V. Lazutin for conducting the tests with low-energy electrons. Figures 13; tables 6; references 19.

Standard Enthalpy of Y_2BaCuO_5 Formation

937D01424 Moscow ZHURNAL FIZICHESKOY KIIMII in Russian Vol 67 No 8, Aug 93 (manuscript received 25 Nov 92) pp 1593-1596

[Article by A.S. Monayenkova, A.A. Popova, N.V. Zaytseva, Yu.Ya. Skolis, L.A. Tiflova, O.V. Boltalina, and M.A. Bykov. Moscow State University imeni M.V. Lomonosov, Department of Chemistry; UDC 536.7]

[Abstract] An experimental study of the Y_2BaCuO_5 compound was made concerning its thermochemical properties, specifically the enthalpy of its formation by reaction with metallic yttrium in 2.19 N HCl solution and in 0.88 N $HClO_4$ solution. Monophase polycrystalline Y_2BaCuO_5 was produced by three-phase synthesis of CuO + (extra pure) + Y_2O_3 (99.99% pure) + $BaCO_3$ (analytically pure). All three ingredients had been first heat treated (Y_2O_3 and CuO at 700°C, $BaCO_3$ at 400°C) before being mechanically mixed in diethyl ether or alcohol until the solvent had completely evaporated. The slurry was molded into 10 mm large pellets, which were then annealed in air at 800°C for 24 h. The pellets were afterwards ground to powder and the latter molded into new pellets, these then being annealed at 900°C. The subsequent three-phase synthesis reaction in each of those two solutions was speeded up by two more grinding-molding-annealing operations, the total annealing time at 900° in the process approaching 150 h. Three batches of independently thus synthesized Y_2BaCuO_5 specimens were examined, x-ray phase analysis and differential scanning calorimetry revealing no residues of Y_2O_3 , CuO , and $BaCO_3$ in them. The specimen of metallic yttrium was found to be admixed with 0.25 wt.% bound oxygen (Y_2O_3), 0.12 % bound nitrogen (YN), 0.023 wt.% bound carbon (YC₂), 0.0001 wt.% bound hydrogen (YH₁), 0.05 wt.% Ta, 0.007 wt.% Cu, 0.006 wt.% Al, 0.005 wt.% Fe, 0.003 wt.% Gd, 0.002 wt.% Si, 0.002 wt.% As, 0.001 wt.% La, less than that but not less than 0.0001 wt.% Cl, Tb, Nd, Nb, Ce, Pr, Er, Ni, Ti each. Measurements were made at 298.15 K temperature. The enthalpy was measured in a hermetic vibratory calorimeter made of titanium and wrapped in an insulating jacket. The temperature was measured with a copper resistance thermometer. The standard enthalpy of Y_2BaCuO_5 formation in either of the two solutions was then calculated on the basis of thus obtained experimental data along with already available data pertaining to those two respective thermochemical reaction cycles. The calculations have yielded an average weighted -(2689+/-6) K kJ/mole, compared with -(59+/-7) kJ/mole enthalpy of the $Y_2O_3 + BaO + CuO \rightarrow Y_2BaCuO_5$ reaction. Tables 2; references 16.

 $YBa_2Cu_3O_{7-x}+25\% Ag$ Behavior During Annealing in Oxygen and Helium

937D0148G Moscow FIZIKA I KHIIMIYA OBRABOTKI MATERIALOV in Russian No 3, May-Jun 93 pp 145-148

[Article by V.L. Arbuzov, A.E. Davletshin, V.R. Poskrebysheva, V.I. Shalayev, V.V. Sagaradze, V.M. Alabyev, Physics of Metals Institute at the Urals Department of Russia's Academy of Sciences, Yekaterinburg; UDC 537.312.62]

[Abstract] The effect of silver additions on the properties of Y-Ba-Cu-O high- T_c ceramics, particularly its beneficial effect on the mechanical and superconducting properties, and its role on the oxygen diffusion and concentration are discussed, and an attempt is made to ascertain the specific role it plays in the oxygen behavior in the ceramic during the annealing of $YBa_2Cu_3O_{7-x}$ HTSC ceramics with different density in various atmospheres. To this end, HTSC ceramics are sintered in the air at a 1,203K temperature for 6 h from powders of the superconducting $YBa_2Cu_3O_{7-x}$ phase with a particle size of 3, 5, 10, and 14 μm . Rectangular samples are cut from the $YBa_2Cu_3O_{7-x}+25\% Ag$ ceramic thus prepared, and their superconducting properties are monitored by measuring the magnetic susceptibility in weak magnetic fields of 0.1 A/m at a 3 kHz frequency. The microstructure of annealed samples is investigated under a scanning electron microscope. The dependence of the critical temperature on the duration of annealing in oxygen and subsequent annealing in helium and the dependence of the critical temperature on the duration of helium annealing of samples annealed in oxygen beforehand are plotted. Two saturation phases—fast and slow—are identified. Additions of silver make it possible to relax stress and thus increase the critical temperature. The findings confirm that the presence of Ag improves the superconducting properties of ceramics and their stability in an inert gas atmosphere. Figures 2; references 12: 5 Russian, 7 Western.

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Effect of Thermal Cycling on $\text{YBa}_2\text{Cu}_3\text{O}_{7-x}$ Ceramics Degradation in Water Vapors and Protective Properties of Hydrocarbon Coat

937D0148H Moscow FIZIKA I KHIMIYA
OPRABOTKI MATERIALOV in Russian
No 3, May-Jun 93 pp 149-151

[Article by V.L. Arbuzov, O.M. Bakunin, V.R. Poskrebysheva, I.Sh. Trakhtenberg, Physics of Metals Institute at the Urals Department of Russia's Academy of Sciences, Yekaterinburg; UDC 537.312.62]

[Abstract] The issues of the high- T_c ceramics stability under operating conditions and bulk and surface degradation due to the high OH^- ion mobility and the ability of water to penetrate the pores and intergranular spaces are addressed, and the effect of multiple thermal cycling from room temperature to 77K of both bare and hydrocarbon coat protected ceramic on its subsequent stability to degradation in water vapors is investigated. To this end, 0.5-1 mm thick disc shaped samples with a 10 mm diameter are cut from a single blank made by a standard powder technology at the pilot plant of the State Scientific Research and Development Institute of the Rare Metal Industry. The samples are protected by applying a hydrocarbon coat ($\text{C}_n\text{H}_{2n+2}$, where $n=30-35$) in a 10 Pa vacuum in an organic solvent. The samples are exposed to five thermal cycles while the sample condition is

monitored by the temperature dependence of the magnetic susceptibility measured by the variable inductance method in an alternating magnetic field. The study shows that thermal cycling enhances degradation of the $\text{YBa}_2\text{Cu}_3\text{O}_{7-x}$ ceramic under the effect of water vapors while the hydrocarbon coat protects the HTSC ceramic from degradation under the experimental conditions; the hydrocarbon coat's protective properties decrease after thermal cycling. Figures 1; references 14: 4 Russian, 10 Western.

Wear Resistance of Ceramic Materials Based on Silicon Carbide and Nitride

937D0144A Kiev POROSHKOVAYA
METALLURGIYA in Russian No 5 (365),
May 93 pp 3-8

[Article by G.G. Gnesin, Materials Science Problems Institute, Kiev; UDC 621.762:621.9.025]

[Abstract] Silicon carbide and nitride (SiC and Si_3N_4) are thermodynamically stable compounds with a primarily covalent type of chemical bonding. They have good mechanical and thermophysical properties and remain hard and strong within a broad temperature range. They are thus promising for use in ceramic materials. A study analyzed the various factors affecting the wear resistance of SiC and Si_3N_4 . One big factor dictating the wear resistance of SiC - and Si_3N_4 -based ceramics that are members of friction couples is the nature of the other member of the couple. In ceramic-metal friction couples the metal may react chemically with SiC and Si_3N_4 and thus alter their properties. Oxide ceramics are more wear resistant than nonoxide ceramics, which indicates the greater chemical activity of SiC and Si_3N_4 in relation to iron and its alloys. Moisture is also important: In the presence of absorbed moisture, SiC and Si_3N_4 form SiOH layers that improve friction conditions to a certain point. Si_3N_4 -based ceramics are among the most promising cutting materials. The thermal stability of nitride ceramic is extremely high and is comparable to that of hard alloys with a ductile metal binder. Optimization of phase composition is critical to increasing the wear resistance of ceramics. Analysis of the various factors affecting wear resistance indicates that adhesion, mechanochemical and diffusion interaction in the contact zone, wear under the effect of fatigue and thermal stresses, and abrasive wear are all critical. A comparison of the wear resistance characteristics of poreless ceramic materials based on SiC and Si_3N_4 indicates that under conditions of maximum energy stress (such as when metal is being cut), dense Si_3N_4 -based ceramics are superior because of their high fracture toughness and thermomechanical strength coupled with their lesser chemical activity. Carbide ceramics, which are highly wear resistant under lower loads, are thus recommended for use in sliding bearings and end seals, where hardness and quality of the material's surface are decisive and

where high heat conduction and a low friction coefficient prevent heating and mechanochemical interaction in the friction zone. SiC-based ceramics thus appear interesting for use under conditions where their surfaces will be subjected to gas- and hydroabrasive flows. Figures 8, table 1; references 11: 7 Russian, 4 Western.

Ultrahard Materials in Machine Building

937D0144B Kiev *POROSHIKOVA*
METALLURGIYA in Russian No 5 (365),
May 93 pp 8-13

[Article by N.V. Novikov, Ultrahard Materials Institute, Ukraine Academy of Sciences, Kiev; UDC 621.762]

[Abstract] In the past 20 years the use of ultrahard materials (including synthetic diamonds and cubic boron nitride) has increased exponentially. About 70 percent of the ultrahard materials produced are used in machine building and metal working. Ukraine's machine building sector uses about 30 types of ultrahard materials and more than 4,500 type sizes of tools made from them. One of the main tasks in retooling the machine building sector is to intensify the processes used to machine construction materials. The strong scientific and production potential that has been created in Ukraine has resulted in the following achievements in machine building: production and use of single 3-mm synthetic diamond crystals for ultraprecision machining; replacement of the low-productivity process of grinding and manual final working by machining with sharp-edge tools made of ultrahard materials so as to achieve high-precision and high-quality products; use of sharp-edge tools to machine new high-strength and heat-resistant ceramic materials; and development of high-productivity and precision abrasive and sharp-edge tools for NC machines, automated lines, machining centers, and FMS. Work to further specialize individual types of ultrahard materials for narrower applications areas (as is

done abroad) is being considered. The Ultrahard Materials Institute in Ukraine has developed a series of ultrahard material-based tribotechnical composites that do not contain scarce tungsten. The new material, which is called tribonit [as published] and contains chromium carbide, nickel, and cubic boron nitride, has been used in the form of bilayer sealing rings in pumps used in oil recovery. New tribotechnical materials have made it possible to create 400-mm-diameter bearings with a continuous working layer of ultrahard material, 700-mm-diameter bearings with a fragmented working layer, and 800-mm-diameter bearings with reinforcing fibers. Other research at the Ultrahard Materials Institute has resulted in the creation of a high-productivity composite tool based on large polycrystals of ultrahard materials for finishing and roughing metals and alloys, a combined tool that can machine several surfaces in a single pass, and a process for laser-shaping profiled inserts made of polycrystalline ultrahard materials. In recent years, a virtually new sector, i.e., the production and use of different types of built-up, plasma, detonation, and other wear-resistant coatings, has made it possible to double and even triple the life of machine components and units. The new ultrahard materials hexanite-R and kiborit [as published] have made it possible to work wear-resistant materials with a hardness of 44-46 HRC or more. In a number of cases, tools made of kiborit make it possible to work at a cutting depth of up to 2.0-2.5 mm, and cutting tools made of hexanite-R may be used at cutting depths up to 0.8-1.0 mm. New tribotechnological methods have made it possible to increase the reliability, durability, and efficiency of many machines and mechanisms. New precision tools have been developed based on synthetic diamonds and cubic boron nitrides that rival the durability of tools made of natural diamonds, and new grinding wheels made of synthetic diamonds and cubic boron nitride based on a metal binder are 3 to 5 times more durable than tools based on a ceramic binder.

Effect of Grain Size and the Concentration of Diamond Powders on the Compaction and Properties of Metal-Diamond Materials

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METALLURGIYA in Russian No 5 (365), May 93
(manuscript received 28 Jan 92) pp 26-28

[Article by V.K. Sorokin, T.M. Kolosova, L.I. Vorobyeva, and M.V. Paryshev, Nizhgorod Polytechnical Institute; UDC 621.762]

[Abstract] A study examined the effect of grain size and the concentration of ASM diamond filler (powder) on the compaction and properties of metal-diamond materials with an Ni-25% Cu-5% Fe binder. The study metal-diamond materials were produced by rolling (porosity, 31-34 percent). After sintering, the sheets of material were subjected to several cycles of cold rolling with intervening cycles of disordering heat treatment. The specimens were studied on a DRON-2 diffractometer. The specimens' lattice period increased steadily after each of three cycles of heat treatment because of the development of diffusion processes of the formation of a ternary solid solution based on nickel oxide; consequently, the specimens' microhardness increased from 1,310 to 1,630 MPa. Increasing the number of diamonds used had a greater effect on the resistivity of sheets 30-60 μm thick than increasing the grain size did. Increasing the concentration of diamond filler from 12.5 to 25 percent by volume resulted in a slight increase in microhardness of the metal binder. The use of hot rolling instead of cold rolling followed by heat treatment resulted in decreases in both microhardness and resistivity. A quadratic regression equation describing the dependence of the tensile strength of sheet material on the concentration and grain size of diamond filler and sheet thickness was derived. Tables 2; references 3 (Russian).

Effect of Low-Temperature Annealing on the Structure and Superconducting Properties of $\text{YBa}_2\text{Cu}_3\text{O}_{7-\delta}$ Compounds With Different Nonstoichiometry With Respect to Oxygen

937D0146E Yekaterinburg FIZIKA METALLOV I METALLOVEDENIYE in Russian Vol 75 No 2, Feb 93 (manuscript received 28 May 92; after revision 24 Aug 92) pp 125-131

[Article by S.V. Sudareva, Ye.I. Kuznetsova, T.P. Krinitsina, I.B. Bobylev, V.N. Morycheva, L.V. Zherdeva, and Ye.P. Romanov, Metal Physics Institute, Ural Department, Russian Academy of Sciences; UDC (546.562+538.945):621.785.375]

[Abstract] A study examined the effect of low-temperature annealing at 200°C for 100 hours on the structure and superconducting properties of $\text{YBa}_2\text{Cu}_3\text{O}_{7-\delta}$. Two series of single crystals measuring 1 x

2 x 0.1 mm with stoichiometric and nonstoichiometric compositions were prepared. The first were grown by the method of dissolution in a melt, with a close-to-eutectic mixture of BaO and CuO serving as the solvent. The melting and crystallization were performed in Y_2O_3 -stabilized zirconium dioxide crucibles. The starting mixture consisted of 44.8 percent BaO, 53.5 percent CuO, and 1.7 percent Y_2O_3 . Before the crystallization, the melt was held at 1,030°C for 4 hours, cooled at a rate of 1°/h from 1,000°C to 880°C, and then further cooled to room temperature at a rate of 100°/h. The single crystals in the second series were subjected to additional oxidative annealing in an oxygen atmosphere at 400-650°C for 26 hours to achieve oxygen stoichiometry. The specimens were studied on a DRON-3M automated system. The low-temperature annealing was conducted at 200°C for 35 and 100 hours. In complete consistency with Khachaturyan's theory, the structure of the single crystals with a stoichiometric composition (i.e., $\text{YBa}_2\text{Cu}_3\text{O}_7$) turned out to be stable and did not undergo any noticeable changes as a result of the low-temperature annealing at 200°C for 100 hours. Their coarse twin structure was completely maintained, their diamagnetic response curve remained virtually unchanged, and their high temperature of transition to a superconducting state prior to annealing remained the same. The single crystals with a nonstoichiometric composition, on the other hand, underwent significant changes after low-temperature annealing for 200 hours. Their coarse twins were replaced by a finely disperse tweed structure, and many differences between their electron-diffraction patterns recorded before and after annealing were evident. Their striation after annealing was attributed to waves of atomic displacements. Figures 7; references 7: 1 Russian, 6 Western.

Volatilization of Radionuclides in Plasmochemical Processing of Radioactive Waste

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OBRABOTKI MATERIALOV in Russian No 4, Jul-Aug 93 (manuscript received 1 Dec 92) pp 74-82

[Article by S.A. Dmitriyev, I.A. Knyazev, S.B. Stefanovskiy, and F.A. Diafanov, Moscow Scientific-Industrial Association "Radon"; UDC 621.039.73]

[Abstract] An experimental study of low-level solid and liquid radioactive waste was made concerning volatilization of the principal radionuclides during its plasmochemical processing. Ash residue from a "Fakel" (Torch) reverberatory furnace heated by fuel plasma burners and incinerating solid radioactive waste, or dry salt residue (mainly NaNO_3 for liquid radioactive waste, was poured into alundum crucibles for melting and further processing. The remaining ingredients (quartz sand, steel chips, FeO) were either mixed with the ash residue or laid on top of it. The crucibles were placed on the hearth of a shaft furnace and heated by fuel plasma burners

operating with a propane-butane fuel mixture and 0.3-0.4 excess air, both fuel and air generating the plasma. Solid radioactive waste with or without flux was thus processed. Liquid radioactive waste was first dried and then vitrified in a uniflow plasmochemical reactor furnace, with the aid of pneumatically injected vitrifying additives (datolite, quartz sand, clay), prior to further processing in the shaft furnace. Specimens of processed material were analyzed in an atomic-absorption spectrometer for chemical composition, in an LP-4900 Nokia spectrometer for β -radiation and γ -radiation, with a ZnS(Tl) detector for α -particle emission, and in a DRON-3M x-ray diffractometer with a CuK α -radiation for phase composition. The principal radionuclides identified were ^{137}Cs , ^{60}Co , ^{90}Sr , ^{144}Ce , and α -particle emitting actinides. Measurements were also made to determine the percentage loss of radionuclides by volatilization and the distribution of remaining radionuclides between silica and metal-composite slag components. The results of the experiment indicate that lowering the basicity of the melt by increasing the silica fraction in the slag will suppress the volatilization of ^{137}Cs and adding metal-composite to the slag will completely inhibit the volatilization of ^{60}Co , while melting radioactive waste under flux reduces the volatilization of all radionuclides. Tables 7; references 11.

Plasmochemical Processing of Solid Radioactive Waste

937D0143E Moscow FIZIKA I KHIKIYA
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Jul-Aug 93 (manuscript received 1 Dec 92) pp 65-73

[Article by S.A. Dmitriyev, S.V. Stefanovskiy, I.A. Knyazev, and F.A. Lisanov, Moscow Scientific-Industrial Association "Radon"; UDC 621.039.73]

[Abstract] An apparatus with a plasma-arc shaft furnace has been designed and built especially for high-temperature processing of low-level and medium-level solid radioactive waste by melting and casting. The shaft is heated from below by two plasma arcs with a fuel-air mixing antechamber. Solid radioactive waste gravity-fed into the shaft through a sluice on top descends through a dryer compartment and then a gasifier compartment into a combustion-pyrolyzer chamber and from here into a crucible. Inorganic residue in the form of liquid slag flows from here into containers underneath where it solidifies, whereupon the ingots are conveyed into another furnace for annealing prior to their transfer into storage. Gases generated in the combustion chamber and

organic residue, mainly resin aerosol produced by pyrolysis, pass successively through the layer of radioactive waste and an afterburner chamber, where almost all the resin is depleted, then through an evaporator (heat exchanger), a metal glove filter, a condenser, a preheater, and finally a fine-scrubbing filter. An exhaust fan blows the clean flue gases into a smoke stack. The condensate descends from the condenser into a container underneath and is from there driven by a pump through a nozzle into that heat exchanger to serve as coolant. The fuel for heating the furnace and the shaft is a mixture of hydrocarbons which together with air generate the plasma. The plasma burner, operating with 0.3-0.4 excess air, generates a 2000-2500 K hot stream of gaseous fuel combustion products (CO, CO₂, H₂) with an ultrahigh concentration of radioactive particles and localized large amounts of energy. A theoretical design and performance analysis for operation at a 2000° temperature has indicated that increasing the shaft diameter will almost proportionally increase the productivity of the apparatus in kg/h of waste processing and that increasing the height of the layer of solid radioactive waste in the shaft will almost proportionally decrease the fraction of radionuclides escaping from the shaft. This was verified by actual operation of the prototype shaft furnace at a 1450°C temperature: a furnace with an 8 m³ capacity and 0.12 m² floor area, its hearth being lined with chromium-magnesite brick and its 3 m high shaft being lined inside with mullite brick. The apparatus was tested on radioactive waste containing materials such as wood, paper, polymers, laboratory animal corpses, ceramic, glass, metal, construction debris, and others. The density of produced slags was found to vary over the 2000-5000 kg/m³ range. Their chemical stability was determined according to the IAEA procedure. Specimens were analyzed in an atomic-absorption spectrometer for chemical composition, in an LP-4900 Nokia γ -spectrometer for radiation emitters, and in a DRON-3M x-ray diffractometer for phase composition. The principal radionuclides thus identified in the slag and their specific activity were: ^{137}Cs (0.5-3 MBq/kg), ^{90}Sr (0.1-1 MBq/kg), ^{59}Fe (20-80 kBq/kg), ^{60}Co (20-80 kBq/kg), and nuclides emitting alpha particles (5-1000 kBq/kg). Their distribution in the slag was measured and they were found to have segregated: ^{137}Cs and ^{90}Sr in the silica, ^{59}Fe and ^{60}Co in metal components and slag was also measured. The slag was also found to contain an excess of MgO, Al₂O₃, Cr₂O₃, and SiO₂ oxides, evidently due to corrosion of the brick linings. The shaft furnace is capable of processing solid radioactive waste at a rate of 500 kg/(m²·s). Figures 5; tables 2; references 12.

Heating of Aluminum Particles with Oxide Sheaths by High-Intensity Laser Radiation

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OBRAZOTKI MATERIALOV in Russian No 4,
Jul-Aug 93 (manuscript received 26 Nov 92) pp 18-27

[Article by L.G. Astafyeva and A.P. Prishivalko, Institute of Physics imeni B.I. Stepanov at the Belarusian Academy of Sciences, Minsk; UDC 536.4232:535.211]

[Abstract] Heating of aluminum particles with oxide coatings by 10.6 μm radiation of a CO_2 laser from 293 K room temperature to the 933 K melting point is analyzed theoretically. The energy distribution inside such a particle is described in the approximation of a double-layer spherical particle consisting of a metal nucleus and an oxide shell. The energy distribution in such a model is calculated according to the theory of diffraction of electromagnetic waves by a multilayer sphere. Noteworthy is the cylindrical symmetry of the energy distribution in such a sphere with respect to its Principal diameter, namely the diameter of the sphere which lies on the axis of the incident laser beam. Noteworthy is also the significant difference between the distributions of absorbed energy over the nucleus (metal) surface and over the outer shell (oxide) surface. The system of two nonlinear two-dimensional (r, θ, t) partial differential equations describing the kinetics of temperature distribution in the aluminum nucleus and in the oxide shell is solved for the appropriate initial and boundary conditions, which can be done only by numerical methods. The coefficient of heat transfer from oxide coating to ambient medium is estimated in accordance with the molecular kinetic theory of gases. The results of calculations made for an aluminum particle with oxide coating in a nitrogen atmosphere indicate that the nonuniformity of heating increases with increasing laser radiation intensity and with increasing particle radius, also that both the heating rate and the temperature reached by such a particle depend largely on the on the thickness of the oxide coating. Figures 5; references 14.

Growing Thin Films of Metal Oxides by Ion-Beam Sputtering

937D0143G Moscow FIZIKA I KHIIMIYA
OBRAZOTKI MATERIALOV in Russian No 4,
Jul-Aug 93 (manuscript received 2 Nov 92) pp 99-104

[Article by A.P. Semenov, N.N. Smirnyagina, V.M. Khaltanova, and A.F. Belyanin, Buryatian Institute of Natural Sciences at Russian Academy of Sciences, Siberian Department; UDC 621.039.531]

[Abstract] Growing thin films of metal oxides (Me_xO_y , $\text{Me}_x\text{Me}_y\text{O}_z$, $\text{Me}_x\text{Me}_y\text{Me}_z\text{O}_w$) by ion-beam sputtering was studied experimentally, the process consisting of three successive stages: 1. pulverization of the metal target(s) under vacuum. 2. transfer of the powder(s) onto a

substrate. 3. buildup of the oxide film of given composition into a desired structure. The dominant mechanism contributing to the film growth is, according to two theories, either the knockout of free metal atoms from the target(s) or the chemical synthesis of first simple and then complex metal oxides on the substrate surface. In the experiment two separate sources of 10 keV argon ions were used for sputtering a PbO target and a MoO_3 target respectively, the two oxide films being deposited on a "cold" (300 K) substrate and then annealed in air. Each ion source was a cathodic plasma of glow discharge in a cold hollow cathode. Subsequent annealing of the films at 723 K enlarged the MoO_3 grains and gradually transformed the tetragonal low-temperature PbO phase into the rhombic high-temperature PbO phase. An intense chemical reaction at 823 K resulted in synthesis of the complex PbMoO_4 oxide, a structure of monophase PbMoO with preferred grain orientation then forming at 923 K. Thin films of $\text{YBa}_2\text{Cu}_3\text{O}_{7-\delta}$ as well as of ZnO and WO_3 films had already been deposited earlier by this process. The results of this study confirm the dominant role of chemical reactions in the film growth, especially when stimulated by addition of heat to the energy of sputtering argon ions. Figures 4; tables 2; references 26.

Principal Trends in Improving Al-Li Alloy Quality for Welded Aircraft Structures

937D0148C Moscow FIZIKA I KHIIMIYA
OBRAZOTKI MATERIALOV in Russian No 3,
May-Jun 93 pp 117-122

[Article by I.N. Fridlyander, A.G. Bratukhin, V.G. Davydov, Scientific Production Association of the All-Union Light Alloys Institute, Moscow; UDC 669.715]

[Abstract] The effect of the quality, reliability, and service life indicator level and stability of aircraft structures on their performance efficiency and the emergence of welded aircraft structures made possible by the development of weldable Al-Li alloy 1420 and its modifications which combine a low density of 2.47 g/cm³, a high modulus of elasticity of 8.8 MPa, a high corrosion resistance, and high specific strength indicators exceeding those of traditional wrought aluminum alloys widely used in the aircraft industry prompted a review of the principal trends of improving the quality of Al-Li alloys for welded aircraft structures. The characteristic properties of alloy 1420 sheets and extruded and stamped semifinished products are summarized, and the properties of stamped samples, ingots produced by flux and flux-free methods, and stamped parts after quenching by various methods are compared. The study demonstrates the efficacy of using Al-Li alloy 1420 for aircraft structures and shows that an optimum combination of mechanical properties and good stress corrosion resistance can be attained by quenching at a specified cooling rate. The use of automatic welding for fuel tank fabrication and the issues of weld quality control and

residual stress monitoring with the help of special methods are addressed. Figures 1; tables 5; references 1.

Controlling Aluminum Alloy Structure by Adding Disperse Silicon Carbide Particles to Melt

937D0148E Moscow *FIZIKA I KHIMIYA OBRABOTKI MATERIALOV* in Russian No 3, May-Jun 93 pp 129-137

[Article by T.A. Chernyshova, A.V. Panfilov, L.I. Kobel'eva, M.I. Tylkina, Metallurgy Institute imeni Baykov at Russia's Academy of Sciences, Moscow; UDC 069.71+546.281]

[Abstract] The outlook for the development of new structural materials, particularly cast metal composites reinforced with disperse refractory oxide, carbide, nitride, and boride particles which are characterized by an elevated high-temperature strength and wear resistance, high tribological properties and dimensional stability, etc., are discussed and it is noted that the composite (KM) properties depend not only on the type and volume fraction of the reinforcing filler but also its distribution in the matrix, the bonding quality, the interphase reaction product amount, composition, and morphology, and the cast matrix structure. The mechanism of the structure and mechanical characteristics' behavior of aluminum alloy with an addition of disperse SiC particles to the melt is investigated. The research materials and methods are outlined; in most cases, the composites were produced by casting into a loam mold or die casting of the matrix melt or composite mixture from the same melt with SiC particles using the V124 hypoeutectoid. The sample composition, the investigation method and exposure duration, the microhardness of the composite and nonreinforced samples, and the chemical content of the composite sample matrices are summarized. The study of the solidification characteristics and mechanism of the V124 matrix alloy in the presence of the reinforcing phase shows that addition of SiC particles to the melt facilitates the development of primary Si crystals in the hypoeutectoid whereby α -Al dendrites form in the melt between the reinforcing particle clusters. The last batches of the melt solidify near SiC particles in the form of a two-component Al-Si or a multicomponent Al-Si-Mg-Cu eutectics. The relationship between the melt structure and thermal properties of SiC particles is established. Figures 4; tables 3; references 12: 6 Russian, 6 Western.

Effect of Hot Deformation and Heat Treatment on Complex Nickel-Based Superalloy Structure Formation

937D0148F Moscow *FIZIKA I KHIMIYA OBRABOTKI MATERIALOV* in Russian No 3, May-Jun 93 pp 138-144

[Article by A.V. Shulga, V.V. Nikishanov, L.G. Kuzmicheva, M.S. Podolskiy, Moscow Engineering

Physics Institute and All-Union Light Alloys Institute, Moscow; UDC 669.24:669.784]

[Abstract] The effect of deformation and heat treatment on the making of semifinished and end products from superalloys which are susceptible to the development of a dendritic structure during solidification and, consequently, dendritic segregation and the effect of various factors on segregation are discussed, and an attempt is made to examine the effect of deformation and heat treatment on the structure formation and C and B distribution patterns in Ni-based high-temperature Ni-Al-Ti-Cr-Co-W-Mo-Nb-C-B alloys which are quite representative of the broad spectrum of today's superalloys. To this end, samples of complex Ni-5Al-2Ti-9Cr-15Co-10W-Mo-2Nb-Fe-C-B alloy after hot upsetting at a 1,100, 1,150, 1,175, and 1,200°C temperature with a reduction of 30, 50, and 70% at a 10^{-3} and 10^{-1} s^{-1} rate are examined. The cylindrical samples are cut from ingots produced by vacuum induction melting with subsequent vacuum arc remelting (VIP+VDR). The Ni-based superalloy flow under straining and the effect of the straining temperature on the boride and carbide phase particle precipitation are plotted. The study makes it possible to establish that hot upsetting of this cast alloy is accompanied by plastic deformation and metal recrystallization in the interaxial segments and dendrite axes while the nonuniform substitutional constituent distribution is responsible for ensuring a nonuniform C and B distribution under all hot upsetting rates. The C and B clustering under straining and subsequent heat treatment is largely determined by the carbide and boride phase precipitation diagram and the effect of plastic deformation and alloy recrystallization on it, i.e., depend on the straining temperature, reduction, and rate. Figures 4; references 9.

Change in the Structure and Mechanical Properties of a Solid Alloy as a Function of the Temperature of Infiltration of the Titanium Carbide Framework by Nickel Alloys

937D0144E Kiev *POROSHKOVAYA METALLURGIYA* in Russian No 5 (365), May 93 (manuscript received 1 Apr 91) pp 70-74

[Article by N.V. Mashkin and O.Yu. Yefimov, Superplasticity of Metals Problems Institute, Russian Academy of Sciences, Ufa; UDC 669.018.95:0.26.2]

[Abstract] A study was conducted to identify the optimal temperature of infiltration of a titanium carbide framework by nickel alloys and to determine the effect of different infiltration temperatures on the structure and mechanical properties of titanium carbide-based composites. Frameworks were produced from titanium carbide powder with an average grain size of 2.9 μm by compaction under a pressure of 200 MPa and vacuum sintering at temperatures of 1,400-1,800°C for 0.5 hours.

The complexly alloyed dispersion-type nickel alloy SNS-1 (i.e., alloyed with cobalt, chromium, molybdenum, tungsten, titanium, aluminum, silicon, and carbon) and the intermetallic-type nickel alloy SNS-2 were used as infiltrants. The infiltration was accomplished by vacuum impregnation at temperatures of 1,350-1,550°C for 0.5 hours. Study specimens were obtained by cutting plates measuring 25 x 8 x 6 mm and then grinding them to 5 x 5 x 7 mm. Three specimen plates were prepared for each sintering and infiltration mode studied. The structure and chemical composition of each specimen was examined under electron and scanning electron microscopes and in an Epikvant automatic structural analyzer. The frameworks' porosities were determined by hydrostatic weighing, and their compression strength was determined on a 1231U-10 tensile testing machine at deformation rates of 0.95 to 1.10 x 10⁻³/s. A 2140TR hardness tester was used to measure their hardness. Structural studies of the titanium carbide frameworks established that at sintering temperatures up to 1,400°C, only point contacts exist between the titanium carbide particles, total porosity reaches 50-60 percent, and the average grain size in the framework equals 3.3 μm. Further increases in sintering temperature are accompanied by an increase in grain size, decrease in pore size, decrease in porosity, and increase in closed porosity. After infiltration of the carbide frameworks by the metal, the alloy's structure consists of two continuous phases: a metal binder and a carbide skeleton. The grains of the carbide skeleton are interconnected through a shell formed as a result of diffusion of the carbide-forming elements from the binder to the matrix. Infiltration at temperatures of 1,330-1,350°C does not result in total filling of the pore space of the titanium carbide frameworks. At 1,380°C, the alloy-forming elements are weakly diffused from the binder and into the carbide grains, and the thickness of the shell of complex carbides amounts to just 0.2-0.4 μm. It increases as the temperature increases, and at 1,450°C it reaches 1-1.5 μm. The residual porosity of the material does not exceed 0.3 percent. The use of the study nickel alloys as infiltrants resulted in composites with superior mechanical properties. Dependences of yield point on infiltration temperature were obtained that may be used in optimizing process parameters. Figures 3, tables 3; references 5: 4 Russian, 1 Western.

Reaction of a Melt of a Mixture of Titanium, Aluminum, and Silicon Oxides With Hot-Compacted Silicon Nitride

937D0144D Kiev POROSHKOVAYA
METALLURGIYA in Russian No 5 (365), May 93
(manuscript received 4 Feb 92) pp 39-44

[Article by V.Ya. Petrovskiy and V.L. Yupko; UDC 532.68]

[Abstract] A study examined the reaction of an Al_2O_3 - TiO_2 - SiO_2 melt with a hot-compacted silicon nitride substrate in a nitrogen medium. The study substrate was obtained by hot compaction of β - Si_3N_4 plus a small amount of Al_2O_3 . The substrates' porosity did not exceed 0.5 percent, and their surface roughness after diamond machining did not exceed 0.1 μm. Before the experiments, the substrates were annealed at 1,750°C for 10 minutes to prevent them from releasing gas during the experiments. The oxide mixture was prepared by wet-mixing commercial products with a purity grading of high. The SiO_2 concentration in the mixture was altered in 10-15 percent increments as the Al_2O_3 : TiO_2 ratio was kept unchanged (so SiO_2 to Al_2O_3 + SiO_2 ratios of 100:0, 70:30, 40:60, 30:70, and 0:100 were used). Two experiments were performed for each oxide mixture. A chemical reaction between the substrate and the melt that caused the melt to harden as the temperature was increased was established. The liquid-phase solidification temperature and wetting angle of the silicon nitride substrate by each of the melt mixtures were plotted on Al_2O_3 - TiO_2 - SiO_2 concentration triangles. The experiments established that both the improvement and worsening of the wetting of the Si_3N_4 crystallites by the liquid phase and solidification of the melt are possible during the heating process. The temperature interval in which the liquid phase exists for real materials amounts to about 15°C, which greatly impedes processes of structure formation involving the liquid phase when Si_3N_4 is sintered. The nitrogen medium in which the oxide mixture was melted was hypothesized to have a significant effect on the mixture's melting point. A surplus of nitrogen in the melting zone and the uneven chemical activity of the oxide mixture at different places on the concentration triangle created a complex wetting angle topography that changes over time. Figures 6; references 7: 3 Russian, 4 Western.

Ferromagnetic Powders Based on Reduced Iron γ -Oxide

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METALLURGIYA in Russian No 6 (366), Jun 93
(manuscript received 14 Feb 91) pp 24-26

[Article by V.V. Nepomnyashchiy, T.V. Mosina, and A.K. Dudchenko, Materials Science Problems Institute, Ukraine Academy of Sciences, Kiev; UDC 621.752:583.221:669.5.17.225]

[Abstract] A study examined the possibility of producing powder permanent magnets from iron-cobalt powders based on a γ -modification of Fe_2O_3 . Study specimens were produced, and their magnetic properties were determined. The specimens were then heat-treated and studied by electron microscope and x-ray diffractometer. The starting iron γ -oxide contained FeO and Fe_2O_4 . After reduction annealing in hydrogen at 400°C, FeO was not detected by roentgenography. Only magnetite

and an α -Fe cubic structure with a lattice parameter of $a_0 = 0.286$ nm was found. Annealing significantly improved all of the specimens' magnetic properties (especially induction of saturation); however, the magnetic properties of the new materials were inferior to analogous materials produced from oxalate and format compounds. Cobalt was then added to the starting material in amounts of 0, 10, 15, 20, and 25 percent by weight. The suspension was dried and roasted in air at 350°C. The dry residue was treated with an acetone solution of oleic acid and ED-20 resin to protect the metal phase from the effects of the environment. After the acetone had been removed, the powder was roasted in hydrogen at 400°C. The new material contained magnetite with a cubic structure, cobalt oxide with a hexagonal structure, and α -Fe with a cubic structure. A highly magnetic phase, evidently Fe-Co alloy, was determined to be present. The addition of cobalt in an amount equal to 20 percent of the weight of the starting suspension resulted in the biggest improvement in magnetic properties: $B_s = 0.78$ T, $B_r = 0.41$ T, $H_s = 88$ kA/m; $W_{max} = 7.5$ kJ/m³, and $\gamma = 4.2$ g/cm³. A destructive layer of organic coating had formed on the surface of the particles, and the particles themselves had a pronounced anisotropic shape. The coatings formed on the particles enabled them to maintain their magnetic properties upon exposure to the environment. Figure 1, table 1; references 8 (Russian).

Production and Selected Properties of Nickel-Mercury Intermetallide

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METALLURGIYA in Russian No 6 (366), Jun 93
(manuscript received 1 Jul 91) pp 65-69

[Article by G.A. Kozbagarova, A.A. Lange, and S.P. Buhman, Chemical Sciences Institute, Republic of Kazakhstan Academy of Sciences, Alma-Ata; UDC 620.197.1]

[Abstract] A study examined the sizes and properties of particles of nickel-mercury intermetallide formed on a mercury cathode by the electrolysis method with $E = -1.3$ V and $T = 50^\circ\text{C}$. The solid phase of amalgams with a nickel content of 1-2 percent by weight, i.e., specimens of NiHg_4 , were studied. The NiHg_4 was in the form of a black powder that remained stable in air for no more than 20 hours. The particles' sizes could not be measured by electron microscopy because of interference by trace quantities of free mercury. Besides containing intermetallide, the powders obtained after the mercury had been distilled off also contained a small (3-4 percent by weight) amount of nickel that probably resulted from partial decomposition of the compound during heating. The said phase was much more stable than the intermetallide (i.e., it lasted at least 2 months as opposed to 20 hours). The powder was made up of aggregates 1,000-2,000 nanometers in size that consisted of spherical

particles with varying degrees of dispersion (from single-digit nanometers to tens and hundreds thereof). The smallest of the aggregates detected were on the order of 5-10 nm in size. Most of the particles were on the order of 35 to 45 nm in size. It was hypothesized that the maximum size of the electrochemically active particles does not exceed 40-50 nm. The electrochemical properties of the solid-phase intermetallide were evaluated. The results refute the published hypothesis that oxidation of the particles of solid-phase metals and intermetallides of amalgams at positive potentials occurs during the inhibiting effect of the process of "dewetting" of the particles, i.e., during the breaking of the mercury film. Instead, the evidence indicated that when the particles of the solid phase of an intermetallide are ionized, the mechanism of the process at the mercury-solution interface does not depend on the medium (i.e., metal or aqueous) in which the solid phase is located. The heterogeneous nickel amalgam studied was thus shown to be a colloidal system with disperse intermetallide particles. Figures 2; references 15: 11 Russian, 4 Western.

Effect of Treatment by Thermal Cycles on the Thermal Transformation Kinetics of Austenite and the Properties of 40N2M Steel

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METALLURGIYA in Russian No 6 (366), Jun 93
(manuscript received 4 Feb 92) pp 47-51

[Article by Yu.G. Gurevich, A.G. Ivashko, V.I. Rakhmanov, and I.F. Pahshin, Kurgan Machine Building Institute; UDC 621.79:621.91:620.181.620.17]

[Abstract] A study examined the effect of treatment by thermal cycles on the mechanical properties and transformation kinetics of austenite under isothermal conditions. The studies were performed on specimens of 40N2M steel with a density of 7,780 kg/m³ and the following chemical components (%): C, 0.39-0.41; Ni, 1.9-2.1; and Mo, 0.2-0.3. PZh2M2 iron powder, PNK-OT2 carbonyl nickel powder, TsMTU molybdenum powder, and GK2 graphite powder served as starting materials. The study specimens, which were produced by hot stamping, were sorbites with a small amount (about 20 percent) of martensite. They were subjected to homogenizing annealing at 1,373 K for 7.2×10^3 seconds. Briquettes measuring 1 x 10 x 50 mm were used for the studies (by the magnetic method) of the transformation. The specimens were austenitized in a melt of $\text{BaCl}_2 + \text{NaCl}$ salts at 1,183 K for 3×10^2 seconds. The first batch of specimens was subjected to isothermal cooling in a melt of tin heated to 523-911 K for 12 minutes. The second batch of specimens was cooled in air from the austenitization temperature to 773-823 K. After five heating-cooling cycles, they were subjected to isothermal treatment in a tin melt at the same temperatures. The beginning of the austenite transformation was assumed to be the point corresponding to a 5 percent austenite

melt, and the end of the transformation was assumed to be the time corresponding to a 95 percent austenite melt. Isothermal diagrams were plotted for the specimens of 40N2M powder steel along with their austenite transformation rates. Isothermal hardening with preliminary treatment by thermal cycles was found to result in a superfine bainite and bainite-martensite structure that in turn resulted in steel with better mechanical properties than that obtained by conventional isothermal treatment. This improvement in mechanical properties was attributed to the fact that an increase in the density of crystalline structure defects promotes an increase in diffusion density of the carbon and alloy-forming elements and in the uniformity of their distribution throughout the material. Figures 3; references 11 (Russian).

Effect of Oxide Coating on Strength Properties of Boron and Silicon Carbide Fibers

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METALLURGIYA in Russian No 6 (366), Jun 93
(manuscript received 6 Dec 91) pp 55-61

[Article by M.Ye. Krison, S.P. Listovnichaya, Yu.L. Pilipovskiy, and Yu.M. Vasilenkov, Materials Science Problems Institute, Ukraine Academy of Sciences, Kiev; UDC 261.763(088.8)]

[Abstract] A study examined the effect of yttrium oxide barrier coatings on the strength properties of γ 100- μ m-diameter boron and silicon carbide fibers. The fibers' tensile strength was measured at room temperature on a Shildik 2038 R-0.05 tensile testing machine. During the tests, tension diagrams in the coordinates' load-relative elongation were plotted for each fiber specimen. The fibers' strength characteristics (i.e., tensile strength, mean-square deviation, and coefficient of variance) were determined by the results of tests of 70-100 specimens. The experimental data were then subjected to statistical processing based on Weibull's distribution. The morphological features of the structures of fibers with coatings and their fracture surfaces were studied by optical and electron microscopy, as well as under an

MIM-7 metallographic microscope. The strength properties of the uncoated fibers varied widely from specimen to specimen. Coatings 1 to 5 μ m thick noticeably reduced the coefficient of variance of their strength. The boron fibers increased in strength when coated with yttrium oxide in a layer up to 1.5 μ m thick (most likely because of the partial dissolution of the boron layer in the most defective places in the structure and the healing of surface microdefects). In the case of the silicon carbide fibers, the dependence of strength on coating thickness corresponded to the statistical characteristics of the distribution. The strength of fibers coated with 1-1.5 μ m yttrium oxide remained at the level of the starting fibers. The yttrium oxide coatings on both types of fibers were found to possess a characteristic bilayer structure consisting of a dense quasi-epitaxial yttrium oxide layer up to 1-1.5 μ m thick and a more crumpling, amorphous outer layer with a maximum thickness of 10-15 μ m. Each batch of test specimens was tentatively divided into two groups depending on the fracture mechanism. The weaker specimens failed as a result of their intrinsic structural defects, in which case the coating was generally completely maintained in the form of a "jacket" and the fracture displayed a single cohesive nature. After the tensile strength tests, the stronger specimens either lost their coating completely or else retained only fragments or "islands" of coating. Morphologic and fractographic studies demonstrated that during the deformation process the microcracks developed on the surface of the oxide layer and gradually spread to the depths of the coating, which eventually peeled off the fiber before it fractured completely. This peeling was attributed to the weak bond between the fiber and the protective layer. The studies established that the concept of "critical thickness" does not have the same significance in the case of yttrium oxide coatings that it has in cases of boron nitride, boron carbide, and silicon carbide coatings. The studies further indicated that two competing forces must be kept in mind when developing protective barrier coatings: The coating must be strong enough to effectively transfer the load from the fibers to the matrix through the interface, and the bond at the interface must not be so strong that it reduces the strength properties of the fibers or composite as a whole. Figures 6; references 8: 3 Russian, 5 Western.

Explosion Welding of Steel Plates at Reduced Mounting Clearances Between Them

937D0148D Moscow *FIZIKA I KHIMIYA OBRABOTKI MATERIALOV* in Russian
No 3, May-Jun 93 pp 123-128

[Article by Yu.L. Alekseyev, G.M. Smirnova, Volgograd Branch of the All-Union Scientific Research Institute of Nuclear Engineering; UDC 621.771.8]

[Abstract] The joint boundary of steel plates after explosion welding at small mounting clearances between them is examined metallographically, and the explosion welding principles are examined. The study shows that a cast layer is dominant on the boundary between the plates after this type of welding and that the layer

character varies depending on the mounting clearance. It is speculated that this fusing is due to the effect of impact-compressed air in the gap between the plates on the plate surfaces. The ammonite detonation rate and free surface behavior are measured, and the free surface behavior of the plate is plotted. The findings demonstrate the principal differences between explosion welding with small and large gaps and show that in the case of small clearances, there is virtually no dynamic effect from the projected plates' collision while setting is due not to the physical solid contact but to the surface fusing as a result of the plasma forming in the gap after the explosion. Thus, products which do not require subsequent machining can be fabricated by cladding large surfaces with thin sheets. Figures 5; references 5.

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